CHEMISTRY SOLUTION STUDY OF GAHARU WATER-MIXTURE

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ABSTRACT

This research is about chemistry solution study of Gaharu-water mixture. The fundamental of chemistry properties of Gaharu-water mixture will be investigated by using Preparative High Performance Liquid Chromatography (PREP-HPLC) and Fourier Transform Infrared Spectroscopy *(*FTIR). This will give benefits for the development of economics and help the engineers provide the accurate properties of Gaharu compound solubility in water. Then, the gaharu marker compound characterized from gaharu water mixture may provide the useful information to engineers and scientists to give an insight to control the gaharu extraction process in a better way. The first objective of the study is to develop the method (protocol) in identify and recognize the Gaharu marker compound in water mixture. The second objective if this research is to identify the compound and molecular chemistry of Gaharu oil via Fourier Transform Infrared Spectroscopy (FTIR) and Preparative High Performance Liquid Chromatography (PREP-HPLC). As the conclusion, Gaharu marker compound that dissolve in the water mixture can be successfully identified by using FTIR and PREP-HPLC. Main marker compound dissolved in water are agarospirol, jinkohol-eremol, jinkohol and kusenol.

KAJIAN KIMIA PENYELESAIAN CAMPURAN GAHARU-AIR

ABSTRAK

Kajian ini adalah mengenai kajian penyelesaian kimia campuran Gaharu-air. Asas sifat kimia campuran Gaharu air akan disiasat dengan menggunakan persiapan Cecair Prestasi Tinggi Chromatography (PREP-HPLC) dan Spektroskopi inframerah transformasi Fourier (FTIR). Ini akan memberi manfaat untuk pembangunan ekonomi dan membantu jurutera untuk mendapatkan sifat kompaun Gaharu yang larut di dalam air dengan tepat. Kemudian, sebatian gaharu penanda yang dicirikan dari campuran air gaharu boleh memberikan maklumat yang berguna kepada jurutera dan saintis untuk mengawal proses pengekstrakan gaharu dengan cara yang lebih baik. Objektif pertama kajian ini adalah untuk membangunkan kaedah (protokol) dalam mengenal pasti sebatian penanda Gaharu dalam campuran air. Objektif kedua kajian ini adalah untuk mengenal pasti kompaun dan kimia molekul minyak Gaharu melalui Spektroskopi inframerah transformasi Fourier (FTIR) dan persiapan Kromatografi Cecair Prestasi Tinggi (PREP-HPLC). Sebagai kesimpulan, Gaharu sebatian penanda yang larut dalam campuran air telah berjaya dikenal pasti dengan menggunakan FTIR dan PREP-HPLC. Sebatian penanda Utama larut dalam air adalah agarospirol, jinkohol-eremol, jinkohol dan kusenol.

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CHAPTER 1

INTRODUCTION

1.1 Introduction

This research aims to gain a better understanding of chemistry solution study of Gaharu-water mixture. Gaharu is the resinous heartwood from Aquilaria trees, which is the native to Southeast Asia and Asia (Frank & James, 2012). In this study, the fundamental of chemistry properties of Gaharu-water mixture will be investigate by using Preparative High Performance Liquid Chromatography (PREP-HPLC) and Fourier Transform Infrared Spectroscopy (FTIR). The analytical method to analyse the gaharu marker compound that dissolved in water mixture will be identified. Thus, this will give benefits for the development of country economics and help the chemical or process engineers provide the accurate properties of gaharu compound solubility in water.

1.2 Problem Statement

This research aims to identify the Gaharu marker compound in water mixture. Basically, water and essential oil will never mix together. When the gaharu oil contacts the surface of the water, it spreads out to form a thin layer. This is because the density of gaharu oil and water are differences. Nevertheless, there are some gaharu marker compounds able to dissolve in the water and their presence can be identified because there is an odour of gaharu in the water after conducting the distillation process. Thus, this research will develop and identify a new analytical method by using Preparative High Liquid Chromatography (PREP-HPLC) and Fourier Transform Infrared Spectroscopy (FTIR) to recognize the marker compound in gaharu-water mixture. Once the marker compound able to be recognized, engineers are able to optimize and purify the gaharu oil from the extraction process.

1.3 Research Objective

At present study, there is a scarcity of chemistry properties of gaharu water mixture from extraction process. Therefore, the study is aimed to achieve several objectives which are as follow:

I. To develop the method (protocol) in identify & recognize the gaharu marker compound in water mixture.

II. To identify the compound and molecular chemistry of Gaharu oil via Fourier Transform Infrared Spectroscopy (FTIR) and Preparative High Liquid Chromatography (PREP-HPLC)

1.4 Scope of the Proposed Study

The scope of this study is to identify the important chemical constituent in Gaharu oil- water mixture from the local Malaysian gaharu essential oil. In addition, this research will investigate the chemistry structure of gaharu water mixture using Fourier Transform Infrared Spectroscopy (FTIR) and Preparative High Performance Liquid Chromatography (PREP-HPLC). The marker compound characterized from gaharu water mixture may provide the useful information to engineers and scientists to control the gaharu extraction process in better and efficient way.

1.5 Expected Outcome

In this research, it is expected that Gaharu marker compound that dissolve in the water mixture can be successfully identified by using FTIR and PREP-HPLC. Main marker compound which possibly dissolve in water are agarospirol, jinkohol-eremol, jinkohol and kusenol. This is because, the sesquiterpene alcohol compound able to form the intermolecular interaction between water molecules. The stronger the intermediate interaction between marker compound-water, the higher the solubility of marker compound in water solvent.

1.6 Significant of Propose Study

This study wills focus on developing and identifying the gaharu marker compound in water mixture. This research will give the benefit for the development of economics and improve the analytical method to analyse the gaharu marker compound that dissolve in the water mixture. In addition, the data can provide an engineer the accurate properties of “gaharu marker compound” solubility in water as solvent during the extraction process. The result would help the local gaharu extractor improve their conventional extraction process to much more efficient techniques.

1.7 Conclusion

In conclusion, this study is about Chemistry Solution Study of Gaharu-Water Mixture. Thus, this research will develop and identify gaharu essential oils that dissolve in the water mixture by using Fourier Transform Infrared Spectroscopy (FTIR) and Preparative High Liquid Chromatography (PREP-HPLC).

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In the world of development, people nowadays are realized the existence of Gaharu in their daily life. Gaharu is the resinous heartwood from Aquilaria trees, which is the native to Southeast Asia and Asia. The trees occasionally become infected with mold and begin to produce an aromatic resin in response to this attack. As the infection grows, it results in a very rich, dark resin within the heartwood. It is this precious resinous wood that is treasured around the world. The resin is commonly called Gaharu, Agarwoods, Oud, Jinko, or Aloeswood and is valued in many cultures for its distinctive fragrance, thus it is used for incense or perfumes.(Frank and James, 2012).

The picture of Gaharu is show in Figure 2.1 in order to get clear understanding about Gaharu.

Figure 2.1 Gaharu Wood (Source: Burhan, 2012)

There are five species of *Aquilaria* (gaharu) were recorded in Peninsular Malaysia,. *Aquilaria malaccensis* is the most popular species among the *Aquilaria* family. There are some chemical components that contribute to the characteristic aroma of Gaharu. Those components are Agarospirol, a-agarofuran, Jinkoh-eremol, 10-epi-g-eudesmol, b-agarofuran, Nor-ketoagarofuran, Kusunol, Jinkohol and Jinkohol II (Surita, 2008).

2.2 Gaharu/ *Malaccensis*

Gaharu or Aquilaria is known under many names based on different cultures. In Malaysia, *Aquilaria malaccensis was* the first agarwood-producing species in 1995. (Lim. & Nooraine, 2010,). Besides, in Chinese is known as Chen-xiang, Jin-Koh in Japanese whereas in Europe it was referred to as Lignum Aquila (eagle-wood) because of the similarity in sound of agila to gaharu. Gaharu can be categorized in many species.

There are five species of *Aquilaria* in Malaysia namely *Aquilaria malaccensis, A.microcarpa, A. hirta,A.rostata and A.beccariana.* Normally, *Aquilaria malaccensis* is the most popular species among the *Aquilaria* family. Gaharu consist of two principles uses namely medicine and perfume.

Gaharu is widely used in traditional medicine as sedative, analgesic and digestive (Alimon et.al, 2011). In China, it is widely used to treat gastralgia, gastric ulcers, gastroptosis, kidney, liver and respiratory problems (Commercial Gaharu Cultivation in Sarawak, 2010). In addition, Gaharu is valued in many types of cultures in all country for its distinctive fragrance and has been widely used as a perfume. Usually, the perfume will come out with resin. The resin looks like a dark to black or a chunk with a fragrant smell if it is burns. Basically, it is found in the heartwood or roots of gaharu producing trees undergoing a chemical and physical change due to the existence of fungus infection. Unfortunately, not all plants can produce resin. (http://www2.thejakartapost.com, 2012).

2.3 Gaharu Extraction

Extraction is a process of obtaining something by chemical or physical or mechanical means from a mixture of compounds (die.net, n.d). There are many types of extraction such as liquid-liquid extraction, and solid phase extraction. Knowing that, liquid-liquid extraction is a separating technology that is based on the distribution of one or more components between two immiscible or almost immiscible liquids. This type of

extraction has many advantages such as very large capacities are possible with minimum energy consumption, heat sensitive products are processed at ambient or moderate temperature and also separation of small contents of high-boiling impurities. Whereas, for solid phase extraction, Solid-phase extraction is an extraction method that uses a solid phase and a liquid phase to isolate one, or one type, of analyte from a solution. It is usually used to clean up a sample before using a chromatographic or other analytical method to quantitate the amount of analyte in the sample (Aldrich, 1998).

2.3.1 Gaharu Essential Oil

Essential oils can be defined as a volatile oil that is usually having the characteristic odor or flavor of the plant from which it is obtained, and used to make perfumes and flavorings (essential oil, 2000). Knowing that, Generally, gaharu oils are mixture of sesquiterpenes,sesquiterpene alcohols, oxygenated compounds, chromone derivatives and resins. Some of the more important compounds are agarospirol, jinkohol-eremol, jinkohol and kusenol that may contribute to the characteristic aroma of gaharu. There are many different of comparisons of chemical at different origin of the Gaharu. The comparison is stated in the Table 2.1.

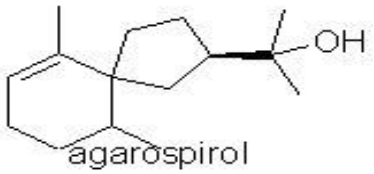
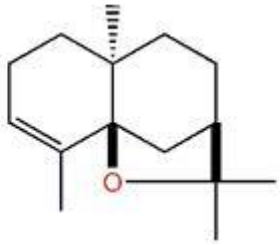
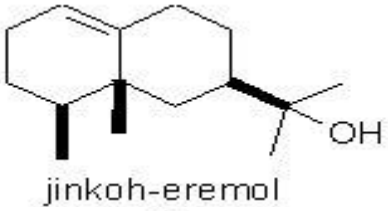
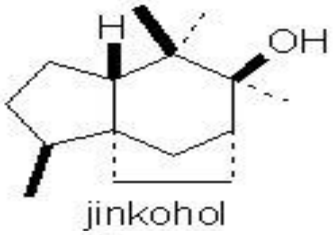
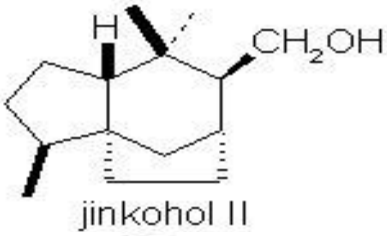
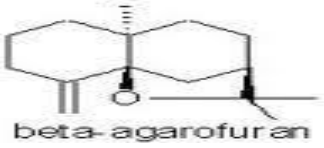
Table 2.1 Comparisons of Chemical at Different Origin of the Gaharu

Gaharu	Chemical components
Type A (A. agallocha)	Agarospirol Jinkoh-eremol Oxo-agarospirol α- and β-agarofuran Dihydroagarofuran Kesunol
Type B (Aquilaria spp.)	Agarospirol Kusunol Jinkoh-eremol Oxo-agarospirol α-agarofuran (-)-10epi-γ-eudesmol Jinkohol Different

(Source: Surita, 2008)

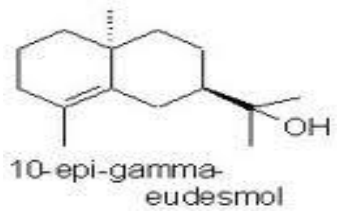
Knowing that, usually different kind of thing will gives different types of its characteristics. Same goes to the Gaharu, Different chemical component in gaharu oil will determine the different characteristic or quality of the gaharu. Thus, for the better and clear understanding, Table 2.2 is tabulated to show some chemical component structure in gaharu essential oil.

Table 2.2 Chemical Component Structures in Gaharu Essential Oil.

Chemical components	Chemical structure
Agarospirol	
a-agarofuran	
Jinkohol-eremol	
Jinkohol	
Jinkohol II	
Beta-agarofuran	

(Source:Surita,2008)

Table 2.2 (Continue)

Chemical Components	Chemical Structure
10-epi-gamma-eudesmol	

People nowadays are realized the importance of using essential oils in their daily life. In the world of development, essential oils are more widely used in modern products than one might expect. Usually the essential oils are extracted by using distillation. They are used to fragrance bathing products, incenses, perfumes and cosmetics. But, in terms of alternative medicine, essential oils are most frequently used in aromatherapy. Same to Gaharu essential oils also, it also used to fragrances, incenses, perfumes and so on.

2.3.2 Gaharu Marker Compound

Gaharu marker compound is the compound that will be used flame photometry to detect certain substances. The main component gaharu marker compounds are agarospirol, jinkohol-eremol, jinkohol and kusenol that may contribute to the characteristic aroma of gaharu. (Adam et.al, 2005). Table 2.3.1 shows the physical and chemical properties of gaharu maker compound.

Table 2.3 Physical and Chemical Properties of Agarospirol

Name of component	Agarospirol
Chemical Formula	$C_{15}H_{26}O$
Molecular Weight	222.366g/mol
Hydrogen Bond Acceptors/donors	Donor
Polarity	Polar

Table 2.4 Physical and Chemical Properties of Jinkohol-Eremol

Name of component	Jinkohol-Eremol
Chemical Formula	$C_{15}H_{26}O$
Molecular Weight	222.366g/mol
Hydrogen Bond Acceptors/donors	donor
Polarity	polar

Table 2.5 Physical and Chemical Properties of Jinkohol

Name of component	Jinkohol
Chemical Formula	$C_{17}H_{14}O_4$
Molecular Weight	250g/mol
Hydrogen Bond Acceptors/donors	donor
Polarity	polar

Table 2.6 Physical and Chemical Properties of Khusenol

Name of component	Khusenol
Chemical Formula	$C_{26}H_{32}O_8$
Molecular Weight	472.527 g/mol
Hydrogen Bond Acceptors/donors	donor
Polarity	polar

2.4 Intermolecular Interaction in Organic Solution

Intermolecular forces are forces of attraction or repulsion which act between neighboring particles such as atoms, molecules or ions. Intermolecular forces are particularly important in terms how molecules interact and form biological organisms or even life. In general, there are four classifications of intermolecular forces such as dipole-dipole forces, van der Waal's forces, Hydrogen bond and Covalent bonding.

2.4.1 Dipole-Dipole Forces

Dipole-dipole interaction is the attraction between a partially negative portion of one molecule and a partially positive portion of a nearby molecule. Dipole-dipole interaction occurs in any polar molecule as determined by molecular geometry. The dipole-dipole force exists in all molecules that are polar. Polar molecules have permanent dipoles that interact with the permanent dipoles of neighboring molecules.

The positive end permanent dipole is attracted to the negative end to another (Figure 2.2).

The dipole-dipole forces much weaker than ionic or covalent bonds and have a significant effect only when the molecules involved are close together (touching or almost touching).

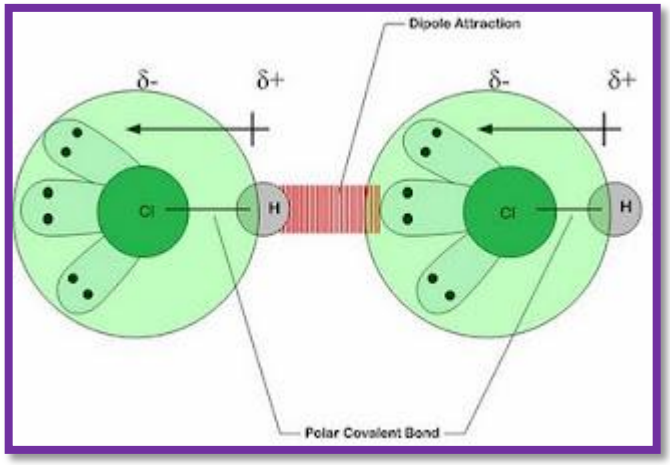


Figure 2.2 Dipole-Dipole Forces (Source: Burhan, 2012)

2.4.2 Van Der Waal's Force

The force arisen from induced dipole and the interaction is weaker than the dipole-dipole interaction. In general, the heavier the molecules, the stronger the van der Waal's force of interaction. The Figure 2.3 shows the van der Wall's force between the molecules. Van der Waals' forces are much weaker than all other types of bonding. They are only significant in atoms and molecules which have no other types of intermolecular forces of attraction.